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IS 1448-98 (1981): Methods of test for petroleum and its products, Part 98: Determination of 1 emulsion stability of emulsifiable cutting oils [PCD 1: Methods of Measurement and Test for Petroleum, Petroleum Products and Lubricants]

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Indian Standard

METHODS OF TEST FOR PETROLEUM AND ITS PRODUCTS

[P : 98]

DETERMINATION OF EMULSION STABILITY OF EMULSIFIABLE CUTTING OILS

(First Reprint NOVEMBER 1991)

UDC 621·892·6 : 543·876

Adapted from the Institute of Petroleum, U. K. Standard No. IP 263/70
(Reapproved 1979)

1. SCOPE

1.1 Cutting oils used in the form of aqueous emulsions should remain stable in use. This method is accordingly designed to assess the stability of cutting oils used in the form of aqueous emulsions.

2. OUTLINE OF THE METHOD

2.1 An emulsion in water of the cutting oil under test is prepared using a magnetic stirrer. The emulsion so prepared is poured into a flask with graduated neck, allowed to stand for a specified time and any separated layer is measured.

3. APPARATUS

3.1 Magnetic Stirrer — with glass or polyethylene jacketed soft iron stirring rod approximately 25 mm long × 6 mm diameter.

3.2 Flask — of 150 ml capacity with a 10 ml graduated neck, having 0·1 ml graduation.

3.3 Hypodermic Syringes — (without needles) of suitable capacity.

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BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

4. MATERIALS

4.1 Synthetic Hard Water — A calcium sulphate solution of total hardness equivalent to 400 ± 10 ppm in terms of calcium carbonate ($CaCO_3$). Prepare by dissolving 0·688 g calcium sulphate $CaSO_4.2H_2O$) per litre of distilled water. Determine the total hardness (and adjust if necessary) by the method described in Appendix A.

5. PREPARATION OF APPARATUS

5.1 Clean the graduated neck flasks and then treat them with chromic acid cleaning solution for at least 2 h. Rinse free from acid with tap water, then several times with distilled water and dry.

> **Caution** — Protective clothing, including eye/face shield should be worn when chromic acid is handled. Splashes on the skin should be immediately and thoroughly washed with water.

6. PROCEDURE

6.1 Make up to eight tests (*see* **6.4.1**) on the oil using preferably the water and the dilution which apply in practice, or failing this use synthetic water at an appropriate dilution.

6.2 Prepare separate amounts of emulsion (*see* **6.2.2**) each sufficient to fill the graduated flask to the upper mark (*see* Note) as follows.

6.2.1 Bring the water and the oil to a temperature 20 ± 4°C before test.

6.2.2 Stir the appropriate volume of water in a 500 ml conical flask, using the magnetic stirrer, at a speed sufficient to create a vortex just deep enough to reach the bottom of the flask. Add the requisite volume of oil quickly from a hypodermic syringe (without needle) of appropriate capacity and continue stirring for 2 minutes after the last drop of oil is added.

NOTE — Enough emulsion shall be prepared to fill the flask to the top mark. Suggested quantities for a selection of water: oil ratios are tabled below:

	EMULSION RATIO							
	5 : 1	10 : 1	15 : 1	20 : 1	25 : 1	30 : 1	50 : 1	80 : 1
Test water, ml	150	200	180	200	200	180	200	240
Oil, ml	30	20	12	10	8	6	4	3

6.3 At once pour enough of the emulsion into the graduated-neck flask to reach the 5 ml calibration mark, stopper, and allow the flask to stand at 40 ± 4°C for 24 h.

NOTE — If marked oil separation is observed immediately on completion of stirring, the test should be discontinued and the fact reported.

6.4 At the end of the 24 h period examine the neck of the flask for separated material (oil, curds, or cream).

Assess whether the emulsion meets the specified level of performance or other specified criteria, based on the measurement of oil and other separation. Classify each test carried out on the emulsion as meeting (PASS) or not meeting (FAIL) the specified level by using the following means.

6.4.1 Carry out up to eight tests on the emulsion according to the following rules.

6.4.1.1 If after five tests there have been five passes then accept the emulsion, but if there are two or more failures reject the emulsion.

6.4.1.2 If after five tests there have been four passes and one failure then carry out three further tests; if and only if these three tests are passes, accept the emulsion; otherwise reject it.

7. REPORT

7.1 Report the volume(s) of (a) oil and (b) total separated material(s) to the nearest 0·1 ml as emulsion stability.

8. PRECISION

8.1 A confidence level of 95 percent has been confirmed by a co-operative programme of tests.

8.1.1 In which the following criteria were used — 'an emulsion was considered as "Pass" if and only if the total separation (oil and cream) was not greater than 0·1 ml after standing 24 h'.

8.1.2 In which the synthetic hard water equivalent to 400 ± 10 ppm of calcium carbonate was used.

8.1.3 For dilution ratios in the range 30 : 1 to 50 : 1.

8.2 This procedure may be invalid if the criteria used allow an emulsion yielding greater than 0·5 ml total separation after 24 h to be marked 'PASS'.

APPENDIX A

(*Clause* 4.1)

METHOD FOR DETERMINATION OF TOTAL HARDNESS OF SYNTHETIC HARD WATER

A-1. SCOPE

A-1.1 This method describes the procedure for the determination of the total hardness of the synthetic hard water specified in **4.1**.

A-2. REAGENTS

A-2.1 Disodium Dihydrogen Ethylenediaminetetra-acetate (EDTA)— 0·02 N. Dissolve 3·72 g of crystalline dihydrate in water and dilute to 1 litre. Check against standard hard water.

A-2.2 Ammonium Buffer — Add 67·5 g of ammonium chloride to 570 ml of ammonia (relative density 0·880) and dilute to 950 ml with water. Dissolve 0·616 g of magnesium sulphate, $MgSO_4.7H_2O$, in 50 ml water and add 0·93 g of solid EDTA reagent. When this is in solution add it to the ammonia and ammonium chloride mixture.

A-2.2.1 The addition of 2 ml of buffer in 100 ml of water is equivalent to the addition of 5 ppm of magnesium in terms of $CaCO_3$ together with its equivalent of EDTA.

A-2.3 Total Hardness Indicator — Weigh 0·5 g of Solochrome Black WDFA or Eriochrome Black T and dissolve in about 2 ml of hot water. Add 10 g of sodium chloride, mix thoroughly, and dry at 105°C. When dry, add 90 g of sodium chloride and 4·5 g hydroxylamine hydrochloride (hydroxyammonium chloride) and grind thoroughly.

NOTE — If desired the 2 ml of water and the 10 g of sodium chloride may be increased provided the total mass of sodium chloride does not exceed 100 g.

A-2.3.1 Alternatively, add 0·5 g of Solochrome Black WDFA or Eriochrome Black T to 100 ml of industrial methylated spirit, warm to dissolve the dyestuff, and add 4·5 g of hydroxylamine hydrochloride (hydroxyammonium chloride). Allow to stand overnight and filter.

NOTE — This indicator will not keep for more than one month.

A-3. PROCEDURE

A-3.1 Transfer 50 ml of the solution to a porcelain casserole, or dish, add 2 ml of ammonium buffer and 0 2 g or 6 drops of total hardness indicator as appropriate. Titrate immediately with EDTA, 0·02 N, until the solution in the casserole has lost all traces of red colour. The colour at the end point is usually pure blue but with some waters a neutral grey end point is obtained (*see* Note).

NOTE — Some substances occasionally present in water samples cause interference with the end point at certain concentrations, while others cause no interference but slightly affect the accuracy of the determinations. The concentrations at which serious interference occurs are higher than are normally found.

A-4. CALCULATION

$$\text{Total hardness, ppm of } CaCO_3 = \frac{\text{ml } 0{\cdot}02\text{N EDTA} \times 1\,000}{\text{ml of solution}}$$

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TO
I S 1448 [P:98] : 1981 METHODS OF TEST FOR PETROLEUM AND ITS PRODUCTS

DETERMINATION OF EMULSION STABILITY OF EMULSIFIABLE CUTTING OILS

(*Page* 1, *clause* **1.1**) — Add the following after **1.1**:

'**1.2** This standard does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.'

(*Page* 2, *clause* **5.1**, '*Caution*') — Read the following in place of the existing:

'PRECAUTIONARY STATEMENTS
Chromic Acid Cleaning Solution

Danger — Causes severe burns. A recognized carcinogen.

Stray oxidizer — Contact with other material may cause fire.

Hygroscopic.

Do not get into eyes, on skin, on clothing.

Avoid breathing vapour of mist.

Keep containers closed.

Use with adequate ventilation.

Do not take internally.

Wash thoroughly after handling.'

(PCD 01)

Reprography Unit, BIS, New Delhi, India